इंटरनेट मानक

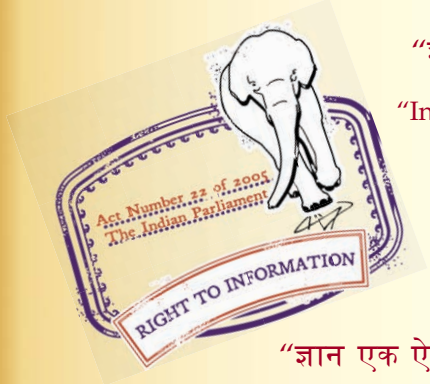
Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 8637 (1986): H Acid [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

IS : 8637 • 1986
(Reaffirmed 2000)

Indian Standard

SPECIFICATION FOR H ACID

(First Revision)

UDC 667·231 : 547·654·2

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

December 1986

Indian Standard

SPECIFICATION FOR H ACID

(First Revision)

Dye Intermediates Sectional Committee, PCDC 11

Chairman	*Representing*
DR P. V. SUBRAMANIAM	Colour-Chem Ltd, Bombay
Members	
SHRI D. K. AGGARWAL	Directorate General of Technical Development, New Delhi
SHRI K. P. DOHARE (*Alternate*)	
DR K. APARAJITHAN	Atic Industries Ltd, Bulsar
SHRI A. M. KAPADIA (*Alternate*)	
SHRI S. D. BHARDWAJ	Ajanta Chemical Industries, New Delhi
SHRI KAPIL DEV (*Alternate*)	
SHRI M. V. DESAI	Atul Products Ltd, Atul
DR J. M. TUREL (*Alternate*)	
DR Y. B. DESAI	Universal Dyestuff Industries Ltd, Vadodara
SHRI HIMAT J. DOSHI	Indian Chemical Manufacturers Association, Calcutta
SHRI V. K. MENON	Amar Dye-Chem Ltd, Bombay
SHRI B. M. NAIK	Indian Dyestuff Industries Ltd, Bombay
SHRI D. G. PATWARDHAN	Development Commissioner, Small Scale Industries, New Delhi
SHRI D. P. SINGH (*Alternate*)	
SHRI S. RAJAGOPALAN	Hindustan Ciba-Geigy Ltd, Bombay
SHRI D. K. MURTHY (*Alternate*)	
SHRI K. L. RATHI	Sudarshan Chemical Industries Ltd, Pune
DR A. THOMAS (*Alternate*)	
DR J. N. SHAH	Sandoz (India) Ltd, Bombay
SHRI K. S. RINDANI (*Alternate*)	
SHRI N. R. TALPADE	Hickson & Dadajee Ltd, Bombay
SHRI M. W. SHENDE (*Alternate*)	
SHRI S. L. TOSHNIWAL	Zenith Steel Pipes (Chemical Division), Bombay
DR V. V. RAO (*Alternate*)	
SHRI V. G. UPADHYE	Synthofine Chemicals of India Pvt Ltd, Bombay
SHRI H. K. VENKATRAMAIAH	Hindustan Organic Chemicals Ltd, Rasayani
SHRI M. S. SAXENA, Director (P & C)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI A. KAR
Joint Director (P & C), ISI

(*Continued on page* 2)

(*Continued from page* 1)

Sulphonic Acids Dye Intermediates Subcommittee, PCDC 11 : 2

Convener	*Representing*
DR S. SIDDHAN	Colour-Chem Ltd, Bombay
Members	
SHRI R. V. BUBNA	Piramal Rasayan Ltd, Bombay
SHRI RAVI ADUKIA (*Alternate*)	
SHRI M. V. DESAI	Atul Products Ltd, Atul
DR J. M. TUREL (*Alternate*)	
DR V. P. KUBBA	Indian Dyestuff Industries Ltd, Vadodara
SHRI K. L. RATHI	Sudarshan Chemicals Industries Ltd, Pune
DR A. S. GULATI (*Alternate*)	
SHRI M. W. SHENDE	Hickson & Dadajee Limited, Bombay
SHRI K. P. RANE (*Alternate*)	
SHRI H. K. VENKATARAMAIAH	Hindustan Organic Chemicals Ltd, Rasayani

Indian Standard

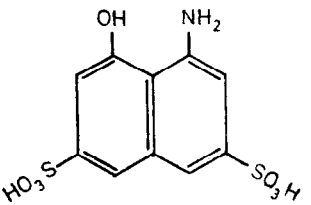
SPECIFICATION FOR H ACID

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 24 March 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 H acid ($C_{10}H_9O_7NS_2$) is an important intermediate used for making azo dyes. It is described as 1-amino-8-naphthol-3, 6-disulphonic acid. It is represented by the following structural formula:



H ACID

(Molecular Mass 319)

0.3 This Standard was first published in 1977. The Committee responsible for the preparation of this standard decided to revise it in order to modify the requirement of matter insoluble in sodium carbonate solution and that of difference between nitrite value and coupling value. The requirement of impurities, namely, Koch's acid content and chromotropic acid content along with their chromatographic test methods have been introduced. The committee envisaged to include the requirement of *W*-acid at a later date when adequate data is made available.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained

*Rules for rounding off numerical values (*revised*).

in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for H acid.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a paste or in the form of light grey to grey lumps or powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR H ACID

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. of Appendix A)
(1)	(2)	(3)	(4)
i)	Assay (by nitrite value), percent by mass (on dry basis), *Min*	75·0	A-2
ii)	Difference between nitrite value and coupling value, percent by mass (on 100 percent basis), *Max*	1·5	A-3
iii)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0·2	A-4
iv)	Chromotropic acid content, percent by mass (on 100 percent basis), *Max*	1·5	A-5
v)	Koch's acid content, percent by mass (on 100 percent basis), *Max*	0·5	A-5

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

*Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Tare, net and gross mass.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of remaining characteristics, namely, difference between nitrite value and coupling value, matter insoluble in sodium carbonate solution, Koch's acid and chromotropic acid content shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirements given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Test shall be carried out according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

*Methods of sampling and test for dye intermediates.

5.2 Quality of Reagents — Unless specified otuherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE 'Pure chemicals' shall mean chemicals that do dot contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR H ACID

A-1. PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

A-2. ASSAY

A-2.1 Reagents

A-2.1.1 *Concentrated Hydrochloric Acid*

A-2.1.2 *Potassium Bromide*

A-2.1.3 *Standard Sodium Nitrite Solution* — 0·1 N.

A-2.1.4 *Potassium Starch Iodide Indicator Papers*

A-2.2 Procedure — Pipette 50 ml aliquot from volumetric solution prepared for the determination of insolubles in sodium carbonate solution (*see* **A-4.2.2**) into a one-litre beaker. Add about 200 ml of water, 35 ml of hydrochloric acid, 5·0 g of potassium bromide and washed-ice. Cool to 10 to 15°C. Titrate, while stirring machanically, with sodium nitrite solution using potassium starch iodide test paper. The end point is reached when a blue-coloured ring appears which can be obtained repeatedly for a period of 10 minutes without further addition of nitrite solution.

*Specification for water for general laboratory use (*second revision*).

A-2.3 Calculation

$$\text{Assay (by nitrite value), percent by mass} = \frac{V_1 \times N_1 \times 319}{M} \text{ (Let this value be } NV \text{)}$$

where

V_1 = volume in ml of standard sodium nitrite solution used in the titration,

N_1 = normality of sodium nitrite solution, and

M = mass in g of the material taken for the test (*see* **A-4.2.1**).

A-3. DIFFERENCE BETWEEN NITRITE VALUE AND COUPLING VALUE

A-3.1 Determination of Coupling Value

A-3.1.1 *Reagents*

A-3.1.1.1 *Sodium carbonate solution* — 20 percent (*m/v*).

A-3.1.1.2 *p-Chloroanilinediazo solution* — 0·1 N.

A-3.1.1.3 *Common salt* — *see* IS : 797-1976*.

A-3.1.2 *Procedure* — Pipette 50 ml aliquot from volumetric solution prepared for the determination of insolubles in sodium carbonate solution (*see* **A-4.2.2**) into a one-litre beaker. Add 100 ml of water, 15 ml of sodium carbonate solution and washed-ice. Cool to 0 to 5°C. Titrate, while stirring mechanically, at 0 to 5°C with aniline diazo solution which is run from a cold water-jacketted burette maintained at 0 to 5°C. Towards the end point, add 50 g of sodium chloride and continue titration until no red colour is obtained at the junction of the coupling solution and the aniline diazo when spotted on a piece of Whatman paper which should be considered as the end point.

NOTE — A yellow colour appears slowly in this region but may be distinguished from the red colour given by H acid in aniline diazo.

A-3.1.3 *Calculation*

$$\text{Coupling value, percent by mass} = \frac{V_2 \times N_2 \times 319}{M}$$

where

V_2 = volume in ml of the *p*-chloroaniline diazo solution used in the titration,

N_2 = normality of the *p*-chloroaniline diazo solution, and

M = mass in g of the material taken for the test (*see* **A-4.2.1**).

*Specification for common salt for chemical industries (*first revision*).

A-3.2 Difference Between Nitrite Value and Coupling Value

Difference between nitrite value and coupling value, percent by mass (on 100 percent basis) $= \frac{D}{NV} \times 100$

where

D = difference between nitrite value and coupling value; and

NV = nitrite value, percent by mass (*see* **A-2.3**).

A-4. DETERMINATION OF MATTER INSOLUBLES IN SODIUM CARBONATE SOLUTION

A-4.1 Reagents

A-4.1.1 *Sodium Carbonate Solution* — 20 percent (*m/v*).

A-4.1.2 *Brilliant Yellow Indicator Papers*

A-4.2 Procedure

A-4.2.1 Weigh accurately about 15 g of the *prepared sample* (*see* **A-1.1**) and transfer to a 500-ml beaker. Paste well with about 100 ml of water. Add sodium carbonate solution till alkaline to brilliant yellow indicator paper. Add about 200 ml of water and heat to dissolve the material completely. Filter hot through counter-poised Whatman filter paper. Wash the residue with hot water till the washings are free from alkali. Dry the residue at 100 ± 5°C to constant mass.

A-4.2.2 Transfer quantitatively the filterate and washing together into a 500-ml volumetric flask and dilute with water up to the mark at room temperature. Mix well.

A-4.3 Calculation

Matter insoluble in sodium carbonate solution, percent by mass $= \frac{m \times 100}{M}$

where

m = mass in g of the residue, and

M = mass in g of the material taken for the test.

A-5. DETERMINATION OF CHROMOTROPIC ACID AND KOCH ACID

A-5.0 Outline of the Method — Chromotropic acid and Koch acid in H acid are determined by ascending paper chromatographic method.

A-5.1 Apparatus

A-5.1.1 *Paper Chromatographic Chamber*

A-5.1.2 *Micropipette* — of 10 µl capacity.

A-5.2 Reagents

A-5.2.1 *Chromatographic Paper* — Whatman No. 3 in the form of a strip of size 25 cm × 16 cm.

A-5.2.2 *Chromotropic Acid* — pure (reference sample). Prepare 0·05 percent, 0·075 percent and 0·1 percent solutions in 1 percent ammonia solution.

A-5.2.3 *Koch Acid* — pure (reference sample). Prepare 0·02 percent, 0·025 percent and 0·03 percent solutions in 1 percent ammonia solution.

A-5.2.4 *Eluent* — *n*-Butanol : Concentrated (36 percent) HCl : water (100 : 45 : 100) (*v/v*).

A-5.2.5 *Spraying Solution* — 1·0 percent Fast Blue B salt solution in water (tetrazotized O-dianisidine zinc double salt).

A-5.3 Procedure

A-5.3.1 Weigh 5·0 g of the sample under test (on 100 percent basis) and dissolve in 100 ml of water and few drops of concentrated ammonia till the solution is clear. Take the chromatographic paper and mark a start line at a distance of 3 cm from any one end of the paper. Spot 10 microlitre of each of the test solution and reference solutions along the start line at a distance of 3 cm from one another.

A-5.3.2 Saturate the chromatographic chamber with the eluent. Suspend the chromatographic strip in the chamber in such a manner that the spotted end of the paper strip just dips into the eluent. The eluent will travel upwards along the strip. Allow it to develop in this manner for 4 hours during which time the solvent front travels approximately 15 cm past the start line. Remove the strip from the chamber, dry it in air.

After drying observe the paper under UV lamp at a wavelength of 254 mm for detecting Koch acid. Compare the intensity of the spot with that of the known standard. Spray the paper with spraying solution and put it in ammonia fumes when the separated spot characteristic of chromotropic acid and unknown are identified by their colour as under:

Content	*Zone*	*Rf Value*	*Colour*
Chromotropic acid	I	0·92	Blue
Unknown	II	0·78	Blue
Koch acid	III	0·52	White fluroescent (under UV lamp at 254 nm)

A-5.4 Report — Estimate the quantities of the impurities by comparing the colour intensities of the spots of the material under test with those of the solutions of the reference samples.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2